

closed 12/20
by JT

MEMORANDUM

TO: Mr. Terry Taylor
Anderson, Mulholland and Associates

DATE: December 18, 2013

FROM: R. Infante *RI*
RE: Data Validation
BMS-ICM, Humacao, PR
BM16.00.09

FILE: JB47485

Accutest Job Numbers: JB47485

SUMMARY

Full validation was performed on the data for six (6) soil samples, one field blank, and one trip blank analyzed selected volatile organic compound by method SW846-8260B. The samples were collected at the BMS-ICM, Humacao, PR site on September 13, 2013 and submitted to Accutest Laboratories of Dayton, New Jersey that analyzed and reported the results under delivery group (SDG) JB47485.

The sample results were assessed according to USEPA data validation guidance documents in the following order of precedence: "USEPA Region 2, SOP HW-24, Validating Volatile Organic Compounds by GC/MS, SW-846 Method 8260B (August 2009-Revision 2), the USEPA National Functional Guidelines for Low Concentration Organic Data Review (August 2009-Revision 2), the USEPA National Functional Guidelines for Organic Data Review for Low Concentration Water (SOP HW-13, August 2009-Revision 3) (noted herein as the "primary guidance documents"). Also, QC criteria from "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods SW-846 (Final Update IV, December 1998)," are utilized. The guidelines were modified to accommodate the non-CLP methodology. The QC criteria and data validation actions listed on the data review worksheets are from the primary guidance document, unless otherwise noted.

In general the data are valid as reported and may be used for decision making purposes. Results in samples JB47485-1 and JB47485-2 were qualified as estimated (J)

SAMPLES

The samples included in the review are listed below

FIELD SAMPLE ID	LABORATORY ID	ANALYSIS
B-3-6(6.5-7)	JB47485-1	VOCs
B-3-6(6.5-7)D	JB47485-2	VOCs
A-3-3(2-2.5)	JB47485-3	VOCs
A-3-3(2-2.5) MSD	JB47485-3D	VOCs
A-3-3(2-2.5) MS	JB47485-3S	VOCs
A-2-6(6.5-7)	JB47485-4	VOCs
FB091313	JB47485-5	VOCs
TB091313	JB47485-6	VOCs

REVIEW ELEMENTS

REVIEW ELEMENTS

Sample data were reviewed for the following parameters, where applicable to the method

- Agreement of analysis conducted with chain of custody (COC) form
- Holding time and sample preservation
- Gas chromatography/mass spectrometry (GC/MS) tunes
- Initial and continuing calibrations
- Method blanks/trip blanks/field blank
- Surrogate spike recovery
- Matrix spike/matrix spike duplicate (MS/MSD) results
- Internal standard performance
- Field duplicate results
- Laboratory control sample/laboratory control sample duplicate (LCS/LCSD) results
- Quantitation limits and sample results

DISCUSSION

Agreement of Analysis Conducted with COC Request

Sample reports corresponded to the analytical request designated on the chain-of-custody.

Holding Times and Sample Preservation

The cooler temperatures were within the QC acceptance criteria of $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$.

Sample preservation was acceptable. Samples analyzed within method recommended holding time.

GC/MS Tunes

The frequency and abundance of bromofluorobenzene (BFB) tunes were within the QC acceptance criteria. All samples were analyzed within the tuning criteria associated with the method.

Initial and Continuing Calibrations

VOCs

The percent relative standard deviations (%RSDs) and response factors (RFs) of all target analytes were within the QC acceptance criteria in the initial calibration. Correlation coefficients (r^2) of target analytes were within the QC acceptance criteria. Ongoing accuracy of the instrument was determined by the analysis of a continuing calibration standard. Initial and continuing calibration meets method performance criteria.

Note: MIBK -20.3 % difference in calibration check; no action taken – professional judgment %D at control limit at two significant figures.

Method Blank/Trip Blank/Field Blank

Target analytes were not detected in laboratory method blanks for VOCs.

One trip/field blanks were analyzed with this data package. No target analytes detected in the trip/field blanks. No equipment blank analyzed as part of this data package.

Surrogate Spike Recovery

The surrogate recoveries were within the laboratory QC acceptance limits in all samples analyzed.

MS/MSD

VOCs

Matrix spike was performed on samples JB47485-3MS/-3MSD; JB47656-7MS/-7MSD; JB46032-13MS/-13MSD; JB47460-4MS/-4MSD; JB48009-1MS/-1MSD; and JB47867-3MS/-3MSD. Recoveries for MS/MSD and RPD were within laboratory control limits except for the followings:

- Ethylbenzene outside control limits in samples JB47485-3MS/-3MSD. No action taken, high level of sample relative to amount spiked.
- MIBK outside control limits in sample JB47460-4MS/-4MSD. MS/MSD criteria apply to the unspiked sample.

Internal Standard Performance

VOCs

Samples were spiked with the method specified internal standard. Internal standard performance met the QC acceptance criteria in all sample analyses.

Field Duplicate Results

Field duplicates analyzed as part of this data set were samples JB47485-1/JB47485-2 (Field-VOCs). RPD results were within laboratory/recommended control limits considering the reporting limits except for the followings:

COMPOUND	SQL	SAMPLE CONC.	DUPLICATE CONC.	RPD	ACTION
Ethylbenzene	410	41000	10000	122	Qualify results in affected samples (J)
Toluene	160	21200	4810	126	
Xylene (total)	220	159000	40600	119	

LCS/LCSD Results

VOCs

The laboratory analyzed one LCS (blank spike) associated with each matrix from this data set. The % recoveries of all spiked analytes were within the laboratory QC acceptance limits.

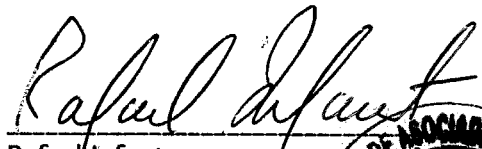
Quantitation Limits and Sample Results

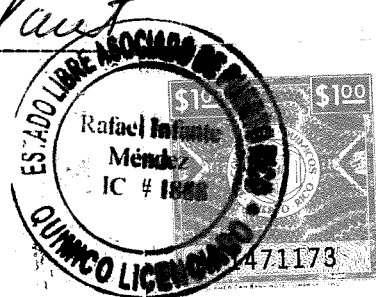
Dilutions were not required with this data set.

Calculations were spot checked.

Certification

The following samples JB47485-1; JB47485-2; JB47485-3; JB47485-3D; JB47485-3S; JB47485-4; JB47485-5; and JB47485-6 were analyzed following standard procedures accepted by regulatory agencies. The quality control requirements met the methods criteria except in the occasions described in this document. The results are valid and can be used for decision taking purposes.


Rafael Infante
Chemist License 1888



Accutest LabLink@769789 12:56 17-Dec-2013

Report of Analysis

Page 1 of 1

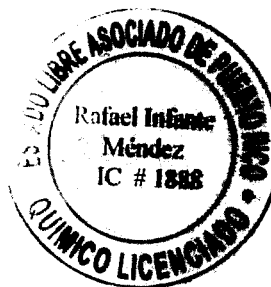
Client Sample ID:	B-3-6(6.5-7)	Date Sampled:	09/13/13
Lab Sample ID:	JB47485-1	Date Received:	09/16/13
Matrix:	SO - Soil	Percent Solids:	80.9
Method:	SW846 8260B SW846 5035		
Project:	BMS-ICM, Humacao, PR		

Run #	File ID	DF	Analyzed	By	Prep Date	Prep Batch	Analytical Batch
Run #1	D213611.D	1	09/26/13	CM	09/16/13 15:00	n/a	VD8724
Run #2	D213434.D	1	09/20/13	CM	09/16/13 15:00	n/a	VD8715

Run #	Initial Weight	Final Volume	Methanol Aliquot
Run #1	6.1 g	5.0 ml	4.0 ul
Run #2	6.1 g	5.0 ml	100 ul

CAS No.	Compound	Result	RL	MDL	Units	Q
100-41-4	Ethylbenzene	41000 J	1600	410	ug/kg	
108-10-1	4-Methyl-2-pentanone(MIBK)	41900	7800	1200	ug/kg	
108-88-3	Toluene	21200 J	1600	160	ug/kg	
1330-20-7	Xylene (total)	159000 J	1600	220	ug/kg	

CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Limits
1868-53-7	Dibromofluoromethane	93%	96%	59-130%
17060-07-0	1,2-Dichloroethane-D4	98%	110%	65-123%
2037-26-5	Toluene-D8	106%	106%	80-124%
460-00-4	4-Bromofluorobenzene	98%	91%	71-132%



ND = Not detected MDL - Method Detection Limit
RL = Reporting Limit
E = Indicates value exceeds calibration range

J = Indicates an estimated value
B = Indicates analyte found in associated method blank
N = Indicates presumptive evidence of a compound

Accutest LabLink@769789 12:56 17-Dec-2013

Report of Analysis

Page 1 of 1

Client Sample ID:	B-3-6(6.5-7)D	Date Sampled:	09/13/13
Lab Sample ID:	JB47485-2	Date Received:	09/16/13
Matrix:	SO - Soil	Percent Solids:	81.8
Method:	SW846 8260B SW846 5035		
Project:	BMS-ICM, Humacao, PR		

Run #	File ID	DF	Analyzed	By	Prep Date	Prep Batch	Analytical Batch
Run #1	D213435.D	1	09/20/13	CM	09/16/13 15:00	n/a	VD8715
Run #2	D213583.D	1	09/26/13	CM	09/16/13 15:00	n/a	VD8723

Run #	Initial Weight	Final Volume	Methanol Aliquot
Run #1	6.2 g	5.0 ml	100 ul
Run #2	6.2 g	5.0 ml	10.0 ul

CAS No.	Compound	Result	RL	MDL	Units	Q
100-41-4	Ethylbenzene	10000	J 60	16	ug/kg	
108-10-1	4-Methyl-2-pentanone(MIBK)	67800 ^a	3000	450	ug/kg	
108-88-3	Toluene	4810	J 60	6.3	ug/kg	
1330-20-7	Xylene (total)	40600 ^a	J 600	84	ug/kg	

CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Limits
1868-53-7	Dibromofluoromethane	96%	97%	59-130%
17060-07-0	1,2-Dichloroethane-D4	108%	104%	65-123%
2037-26-5	Toluene-D8	104%	106%	80-124%
460-00-4	4-Bromofluorobenzene	92%	95%	71-132%

(a) Result is from Run# 2



ND = Not detected MDL - Method Detection Limit
 RL = Reporting Limit
 E = Indicates value exceeds calibration range

J = Indicates an estimated value
 B = Indicates analyte found in associated method blank
 N = Indicates presumptive evidence of a compound

Accutest LabLink@769789 12:56 17-Dec-2013

Report of Analysis

Page 1 of 1

Client Sample ID:	A-3-3(2-2.5)	Date Sampled:	09/13/13
Lab Sample ID:	JB47485-3	Date Received:	09/16/13
Matrix:	SO - Soil	Percent Solids:	80.4
Method:	SW846 8260B SW846 5035		
Project:	BMS-ICM, Humacao, PR		

Run #	File ID	DF	Analyzed	By	Prep Date	Prep Batch	Analytical Batch
Run #1	D213540.D	1	09/25/13	CM	09/16/13 15:00	n/a	VD8721
Run #2 ^a	D213433.D	1	09/20/13	CM	09/16/13 15:00	n/a	VD8715

Run #	Initial Weight	Final Volume	Methanol Aliquot
Run #1	6.1 g	5.0 ml	10.0 ul
Run #2	6.1 g	5.0 ml	100 ul

CAS No.	Compound	Result	RL	MDL	Units	Q
100-41-4	Ethylbenzene	20100	630	170	ug/kg	
1330-20-7	Xylene (total)	140000	630	88	ug/kg	

CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Limits
1868-53-7	Dibromofluoromethane	94%	95%	59-130%
17060-07-0	1,2-Dichloroethane-D4	101%	107%	65-123%
2037-26-5	Toluene-D8	106%	104%	80-124%
460-00-4	4-Bromofluorobenzene	104%	93%	71-132%

(a) Confirmation run.



ND = Not detected MDL - Method Detection Limit
 RL = Reporting Limit
 E = Indicates value exceeds calibration range

J = Indicates an estimated value
 B = Indicates analyte found in associated method blank
 N = Indicates presumptive evidence of a compound

Accutest LabLink@769789 12:56 17-Dec-2013

Report of Analysis

Page 1 of 1

Client Sample ID:	A-2-6(6.5-7)	Date Sampled:	09/13/13
Lab Sample ID:	JB47485-4	Date Received:	09/16/13
Matrix:	SO - Soil	Percent Solids:	80.1
Method:	SW846 8260B SW846 5035		
Project:	BMS-ICM, Humacao, PR		

Run #	File ID	DF	Analyzed	By	Prep Date	Prep Batch	Analytical Batch
Run #1	D213612.D	1	09/26/13	CM	09/16/13 15:00	n/a	VD8724
Run #2	D213436.D	1	09/20/13	CM	09/16/13 15:00	n/a	VD8715

Run #	Initial Weight	Final Volume	Methanol Aliquot
Run #1	6.0 g	5.0 ml	2.0 ul
Run #2	6.0 g	5.0 ml	100 ul

CAS No.	Compound	Result	RL	MDL	Units	Q
100-41-4	Ethylbenzene	83800	3200	850	ug/kg	
1330-20-7	Xylene (total)	331000	3200	450	ug/kg	

CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Limits
1868-53-7	Dibromofluoromethane	95%	96%	59-130%
17060-07-0	1,2-Dichloroethane-D4	100%	109%	65-123%
2037-26-5	Toluene-D8	106%	105%	80-124%
460-00-4	4-Bromofluorobenzene	99%	93%	71-132%



ND = Not detected MDL - Method Detection Limit
RL = Reporting Limit
E = Indicates value exceeds calibration range

J = Indicates an estimated value
B = Indicates analyte found in associated method blank
N = Indicates presumptive evidence of a compound

Accutest LabLink@769789 12:56 17-Dec-2013

Report of Analysis

Page 1 of 1

Client Sample ID:	FB091313	Date Sampled:	09/13/13
Lab Sample ID:	JB47485-5	Date Received:	09/16/13
Matrix:	AQ - Field Blank Soil	Percent Solids:	n/a
Method:	SW846 8260B		
Project:	BMS-ICM, Humacao, PR		

Run #	File ID	DF	Analyzed	By	Prep Date	Prep Batch	Analytical Batch
Run #1	2C111943.D	1	09/25/13	DR	n/a	n/a	V2C5151
Run #2							

Run #	Purge Volume
Run #1	5.0 ml
Run #2	

CAS No.	Compound	Result	RL	MDL	Units	Q
100-41-4	Ethylbenzene	ND	1.0	0.23	ug/l	
108-10-1	4-Methyl-2-pentanone(MIBK)	ND	5.0	0.83	ug/l	
108-88-3	Toluene	ND	1.0	0.23	ug/l	
1330-20-7	Xylene (total)	ND	1.0	0.24	ug/l	

CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Limits
1868-53-7	Dibromofluoromethane	105%		79-117%
17060-07-0	1,2-Dichloroethane-D4	108%		72-123%
2037-26-5	Toluene-D8	109%		82-118%
460-00-4	4-Bromofluorobenzene	108%		75-118%



ND = Not detected MDL - Method Detection Limit
RL = Reporting Limit
E = Indicates value exceeds calibration range

J = Indicates an estimated value
B = Indicates analyte found in associated method blank
N = Indicates presumptive evidence of a compound

Accutest LabLink@769789 12:56 17-Dec-2013

Report of Analysis

Page 1 of 1

Client Sample ID: TB091313
 Lab Sample ID: JB47485-6
 Matrix: AQ - Trip Blank Soil
 Method: SW846 8260B
 Project: BMS-ICM, Humacao, PR

Date Sampled: 09/13/13
 Date Received: 09/16/13
 Percent Solids: n/a

Run #	File ID	DF	Analyzed	By	Prep Date	Prep Batch	Analytical Batch
Run #1	2C111920.D	1	09/25/13	DR	n/a	n/a	V2C5149
Run #2							

Run #	Purge Volume
Run #1	5.0 ml
Run #2	

CAS No.	Compound	Result	RL	MDL	Units	Q
100-41-4	Ethylbenzene	ND	1.0	0.23	ug/l	
108-10-1	4-Methyl-2-pentanone(MIBK)	ND	5.0	0.83	ug/l	
108-88-3	Toluene	ND	1.0	0.23	ug/l	
1330-20-7	Xylene (total)	ND	1.0	0.24	ug/l	

CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Limits
1868-53-7	Dibromofluoromethane	106%		79-117%
17060-07-0	1,2-Dichloroethane-D4	107%		72-123%
2037-26-5	Toluene-D8	109%		82-118%
460-00-4	4-Bromofluorobenzene	106%		75-118%



ND = Not detected MDL - Method Detection Limit
 RL = Reporting Limit
 E = Indicates value exceeds calibration range

J = Indicates an estimated value
 B = Indicates analyte found in associated method blank
 N = Indicates presumptive evidence of a compound

Matrix Spike/Matrix Spike Duplicate Summary

Page 1 of 1

Job Number: JB47485

Account: AMANYWP Anderson, Mulholland & Associates

Project: BMS-ICM, Humacao, PR

Sample	File ID	DF	Analyzed	By	Prep Date	Prep Batch	Analytical Batch
JB47485-3MS	D213430.D	1	09/20/13	CM	n/a	n/a	VD8715
JB47485-3MSD	D213431.D	1	09/20/13	CM	n/a	n/a	VD8715
JB47485-3 ^a	D213433.D	1	09/20/13	CM	n/a	n/a	VD8715

The QC reported here applies to the following samples:

Method: SW846 8260B

JB47485-1, JB47485-2, JB47485-4

CAS No.	Compound	JB47485-3 ug/kg	Spike Q	ug/kg	MS ug/kg	MS %	MSD ug/kg	MSD %	RPD	Limits Rec/RPD
100-41-4	Ethylbenzene	19600	E	3160	18000	-51* ^b	18600	-32* ^b	3	27-139/29
108-88-3	Toluene	1180		3160	3690	79	3720	80	1	32-137/28

CAS No.	Surrogate Recoveries	MS	MSD	JB47485-3	Limits
1868-53-7	Dibromofluoromethane	97%	100%	95%	59-130%
17060-07-0	1,2-Dichloroethane-D4	107%	110%	107%	65-123%
2037-26-5	Toluene-D8	104%	102%	104%	80-124%
460-00-4	4-Bromofluorobenzene	89%	90%	93%	71-132%

(a) Confirmation run.

(b) Outside control limits due to high level in sample relative to spike amount.



* = Outside of Control Limits.



9763 3037 5173 PAGE 1 OF 1 *pw*

FD-36 (Rev. 5-22-64)	Class. Order Control #
<i>ma</i> 9763 3037 5173	
Account Queue #	Account Job # <i>10 1171105</i>

File # Tracking # 8763 3039 5162 Route Order Control #
 Account Control # Account Job # 117105

[illegible]

3A

pu

JB47485: Chain of Custody

Page 1 of 3

DATA REVIEW WORKSHEETS

Project Number: JB47485

Date: 09/13/2013

REVIEW OF VOLATILE ORGANIC PACKAGE

The following guidelines for evaluating volatile organics were created to delineate required validation actions. This document will assist the reviewer in using professional judgment to make more informed decision and in better serving the needs of the data users. The sample results were assessed according to USEPA data validation guidance documents in the following order of precedence: USEPA Region 2, SOP HW-24, Standard Operating Procedure for the Validation of Organic Data Acquired using SW-846 Method 8260B (August, 2009-Revision 2), the USEPA National Functional Guidelines for Low/Medium Concentration Organic Data Review (SOW SOM01.2 SOP HW-33, August 2009 – Revision 2), the USEPA National Functional Guidelines for Organic Data Review for Low Concentration Water (SOP HW-13, August, 2009-Revision 3). Also, QC criteria from "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods SW-846 (Final Update III, December 1996)," specifically for Methods 8000/8260B are utilized. The QC criteria and data validation actions listed on the data review worksheets are from the primary guidance document, unless otherwise noted.

The hardcopied (laboratory name) Accutest data package received has been reviewed and the quality control and performance data summarized. The data review for VOCs included:

Lab. Project/SDG No.: JB47485

Sample matrix: Solid

No. of Samples: 8

Trip blank No.: JB47485-6

Field blank No.: JB47485-5

Equipment blank No.: _____

Field duplicate No.: JB47485-1/-2

☒ Data Completeness

☒ Laboratory Control Spikes

☒ Holding Times

☒ Field Duplicates

☒ GC/MS Tuning

☒ Calibrations

☒ Internal Standard Performance

☒ Compound Identifications

☒ Blanks

☒ Compound Quantitation

☒ Surrogate Recoveries

☒ Quantitation Limits

☒ Matrix Spike/Matrix Spike Duplicate

Overall Comments: Selected VOC's by SW846-8260B

Definition of Qualifiers:

J- Estimated results

U- Compound not detected

R- Rejected data

UJ- Estimated non-detect

Reviewer: Rafael Infante

Date: 12/17/2013

DATA REVIEW WORKSHEETS

DATA COMPLETENESS

MISSING INFORMATION

DATE LAB. CONTACTED

DATE RECEIVED

This image shows a single sheet of white paper with horizontal ruling lines. The lines are evenly spaced and run across the width of the page. There are no margins, text, or other markings on the paper.

DATA REVIEW WORKSHEETS

All criteria were met X
Criteria were not met
and/or see below

HOLDING TIMES

The objective of this parameter is to ascertain the validity of the results based on the holding time of the sample from time of collection to the time of analysis.

Complete table for all samples and note the analysis and/or preservation not within criteria

SAMPLE ID	DATE SAMPLED	DATE ANALYZED	pH	ACTION
All samples analyzed within the recommended method holding time				

Criteria

Aqueous samples – 14 days from sample collection for preserved samples ($\text{pH} \leq 2$, 4°C), no air bubbles.

Aqueous samples – 7 days from sample collection for unpreserved samples, 4°C , no air bubbles.

Soil samples- 7 days from sample collection.

Cooler temperature (Criteria: $4 \pm 2^{\circ}\text{C}$): 3°C - OK

Actions

If the VOCs vial(s) have air bubbles, estimate positive results (J) and reject nondetects (R).

If the % solids of soil samples is 10-50%, estimate positive results (J) and nondetects (UJ).

If the % solid of soil samples is $< 10\%$, estimate positive results (J) and reject nondetects (R).

If holding times are exceeded but < 14 days beyond criteria, estimate positive results (J) and nondetects (UJ).

If holding times are exceeded but < 28 days beyond criteria, estimate positive results (J) and reject nondetects (R).

If holding times are grossly exceeded (> 28 days beyond criteria), reject all results (R).

If samples were not iced or if the ice were melted ($> 10^{\circ}\text{C}$), estimate positive results (J) and nondetects (UJ).

DATA REVIEW WORKSHEETS

All criteria were met X
Criteria were not met see below _____

GC/MS TUNING

The assessment of the tuning results is to determine if the sample instrumentation is within the standard tuning QC limits

X The BFB performance results were reviewed and found to be within the specified criteria.

 X BFB tuning was performed for every 12 hours of sample analysis.

If no, use professional judgment to determine whether the associated data should be accepted, qualified or rejected.

List the samples affected:

If mass calibration is in error, all associated data are rejected.

DATA REVIEW WORKSHEETS

All criteria were met X
 Criteria were not met
 and/or see below

CALIBRATION VERIFICATION

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing and maintaining acceptable quantitative data.

Date of initial calibration: 09/17/13
 Dates of continuing calibration: 09/25/13
 Instrument ID numbers: GCMS2C
 Matrix/Level: Aqueous/low

DATE	LAB ID#	FILE	CRITERIA OUT RFs, %RSD, %D, r	COMPOUND	SAMPLES AFFECTED
Initial and continuing calibration meet method specific requirements					

Criteria

All RFs must be > 0.05 regardless of method requirements for SPCC.
 All %RSD must be $\leq 15\%$ regardless of method requirements for CCC.
 All %Ds must be $\leq 20\%$ regardless of method requirements for CCC.
 It should be noted that Region 2 SOP HW-24 does not specify criterion for the curve correlation coefficient (r). A limit for r of ≥ 0.995 has therefore been utilized as professional judgment.

Actions

If any compound has an initial RF or a continuing RF of < 0.05 , estimate positive results (J) and reject nondetects (R), regardless of method requirements.
 If any compound has a %RSD $> 15\%$, estimate positive results (J) and use professional judgment to qualify nondetects.
 If any compound has a %RSD $> 90\%$, estimate positive results (J) and reject nondetects (R).
 If any compound has a % D $> 20\%$, estimate positive results (J) and reject nondetects (R).
 If any compound has a % D $> 20\%$, estimate positive results (J) and nondetects (UJ).
 If any compound has a % D $> 90\%$, estimate positive results (J) and reject nondetects (R).
 If any compound has $r > 0.995$, estimate positive results and nondetects.

A separate worksheet should be filled for each initial curve

DATA REVIEW WORKSHEETS

All criteria were met X
 Criteria were not met
 and/or see below

CALIBRATION VERIFICATION

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing and maintaining acceptable quantitative data.

Date of initial calibration: 08/29/13
 Dates of continuing calibration: 09/20/13; 09/24/13; 09/25/13; 09/26/13
 Instrument ID numbers: GCMSD
 Matrix/Level: Aqueous/low

DATE	LAB ID#	FILE	CRITERIA OUT RFs, %RSD, %D, r	COMPOUND	SAMPLES AFFECTED
Initial and continuing calibration meet method specific requirements					
09/24/13	cc8686-50		- 20.3 %	4-methyl-2-pentanone	No action taken – professional judgment %D at control limit at two significant figures

Criteria

All RFs must be > 0.05 regardless of method requirements for SPCC.

All %RSD must be $\leq 15\%$ regardless of method requirements for CCC.

All %Ds must be $\leq 20\%$ regardless of method requirements for CCC.

It should be noted that Region 2 SOP HW-24 does not specify criterion for the curve correlation coefficient (r). A limit for r of ≥ 0.995 has therefore been utilized as professional judgment.

Actions

If any compound has an initial RF or a continuing RF of < 0.05 , estimate positive results (J) and reject nondetects (R), regardless of method requirements.

If any compound has a %RSD $> 15\%$, estimate positive results (J) and use professional judgment to qualify nondetects.

If any compound has a %RSD $> 90\%$, estimate positive results (J) and reject nondetects (R).

If any compound has a % D $> 20\%$, estimate positive results (J) and reject nondetects (R).

If any compound has a % D $> 20\%$, estimate positive results (J) and nondetects (UJ).

If any compound has a % D $> 90\%$, estimate positive results (J) and reject nondetects (R).

If any compound has r > 0.995 , estimate positive results and nondetects.

A separate worksheet should be filled for each initial curve

DATA REVIEW WORKSHEETS

All criteria were met X
Criteria were not met
and/or see below _____

V A. BLANK ANALYSIS RESULTS (Sections 1 & 2)

The assessment of the blank analysis results is to determine the existence and magnitude of contamination problems. The criteria for evaluation of blanks apply only to blanks associated with the samples, including trip, equipment, and laboratory blanks. If problems with any blanks exist, all data associated with the case must be carefully evaluated to determine whether or not there is an inherent variability in the data for the case, or if the problem is an isolated occurrence not affecting other data.

List the contamination in the blanks below. High and low levels blanks must be treated separately.

Laboratory blanks

DATE ANALYZED	LAB ID	LEVEL/MATRIX	COMPOUND	CONCENTRATION UNITS
	All_method_blank_meeth_method_specific_criteria			

Field/Equipment/Trip blank

[illegible]

DATA REVIEW WORKSHEETS

All criteria were met X
 Criteria were not met
 and/or see below

V B. BLANK ANALYSIS RESULTS (Section 3)

Blank Actions

Action Levels (ALs) should be based upon the highest concentration of contaminant determined in any blank. Do not qualify any blank with another blank. The ALs for samples which have been diluted should be corrected for the sample dilution factor and/or % moisture, where applicable. No positive sample results should be reported unless the concentration of the compound in the samples exceeds the ALs:

ALs = 10x the amount of common contaminants (methylene chloride, acetone, 2-butanone, and toluene)

ALs = 5x for any other compounds

Specific actions are as follows:

If the concentration is < sample quantitation limit (SQL) and \leq AL, report the compound as not detected (U) at the SQL.

If the concentration is \geq SQL but \leq AL, report the compound as not detected (U) at the reported concentration.

If the concentration is \geq SQL and $>$ AL, report the concentration unqualified.

Notes:

High and low level blanks must be treated separately

Compounds qualified "U" for blank contamination are still considered "hits" when qualifying for calibration criteria.

CONTAMINATION SOURCE/LEVEL	COMPOUND	CONC/UNITS	AL/UNITS	SQL	AFFECTED SAMPLES

DATA REVIEW WORKSHEETS

All criteria were met X
 Criteria were not met
 and/or see below

SURROGATE SPIKE RECOVERIES

Laboratory performance of individual samples is established by evaluation of surrogate spike recoveries. All samples are spiked with surrogate compounds prior to sample analysis. The accuracy of the analysis is measured by the surrogate percent recovery. Since the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the validation of data is frequently subjective and demands analytical experience and professional judgment.

List the percent recoveries (%Rs) which do not meet the criteria for surrogate recovery.

Matrix: solid/aqueous

SAMPLE ID	SURROGATE COMPOUND				ACTION
	1,2-DCA	DBFM	TOL-d8	BFB	

All surrogate recoveries within laboratory control limits

QC Limits* (Aqueous)

_____ LL to UL _____ to _____ to _____ to _____ to _____

QC Limits* (Solid-Low)

_____ LL to UL _____ to _____ to _____ to _____ to _____

QC Limits* (Solid-Med)

_____ LL to UL _____ to _____ to _____ to _____ to _____

1,2-DCA = 1,2-Dichloromethane-d4

TOL-d8 = Toluene-d8

DBFM = Dibromofluoromethane

BFB = Bromofluorobenzene

* QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.

* If QC limits are not available, use limits of 80 – 120 % for aqueous and 70 – 130 % for solid samples.

Actions:

QUALITY	%R < 10%	%R = 10% - LL	%R > UL
Positive results	J	J	J
Nondetects results	R	UJ	Accept

Surrogate action should be applied:

If one or more surrogate in the VOC fraction is out of specification, but has a recovery of > 10%.

If any one surrogate in a fraction shows < 10 % recovery.

All criteria were met X
 Criteria were not met
 and/or see below

VII. A MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD)

This data is generated to determine long term precision and accuracy in the analytical method for various matrices. This data alone cannot be used to evaluate the precision and accuracy of individual samples. If any % R in the MS or MSD falls outside the designated range, the reviewer should determine if there are matrix effects, i.e. LCS data are within the QC limits but MS/MSD data are outside QC limit.

1. MS/MSD Recoveries and Precision Criteria

The laboratory should use one MS and a duplicate analysis of an unspiked field sample if target analytes are expected in the sample. If target analytes are not expected, MS/MSD should be analyzed.

List the %Rs, RPD of the compounds which do not meet the criteria.

Sample ID: JB47485-3MS/-3MSD Matrix/Level: Solid
 Sample ID: JB47656-7MS/-7MSD Matrix/Level: Aqueous
 Sample ID: JB47485-13MS/-13MSD Matrix/Level: Solid

MS OR MSD	COMPOUND	% R	RPD	QC LIMITS	ACTION
<u>JB47485-3MS/-3MSD</u>					
<u>MS/MSD</u>	<u>Ethylbenzene</u>	<u>- 51/-32 %</u>		<u>27-139</u>	<u>No action</u>

Note: No action – high level of sample relative to spike amount.

- * QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.
- * If QC limits are not available, use limits of 70 – 130 %.

Actions:

QUALITY	%R < LL	%R > UL
Positive results	J	J
Nondetects results	R	Accept

MS/MSD criteria apply only to the unspiked sample, its dilutions, and the associated MS/MSD samples:

If the % R for the affected compounds were < LL (or 70 %), qualify positive results (J) and nondetects (UJ).

If the % R for the affected compounds were > UL (or 130 %), only qualify positive results (J).

If 25 % or more of all MS/MSD %R were < LL (or 70 %) or if two or more MS/MSD %Rs were < 10%, qualify all positive results (J) and reject nondetects (R).

A separate worksheet should be used for each MS/MSD pair.

All criteria were met X
 Criteria were not met
 and/or see below

VII. A MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD)

This data is generated to determine long term precision and accuracy in the analytical method for various matrices. This data alone cannot be used to evaluate the precision and accuracy of individual samples. If any % R in the MS or MSD falls outside the designated range, the reviewer should determine if there are matrix effects, i.e. LCS data are within the QC limits but MS/MSD data are outside QC limit.

1. MS/MSD Recoveries and Precision Criteria

The laboratory should use one MS and a duplicate analysis of an unspiked field sample if target analytes are expected in the sample. If target analytes are not expected, MS/MSD should be analyzed.

List the %Rs, RPD of the compounds which do not meet the criteria.

Sample ID: <u>JB47460-4MS/-4MSD</u>	Matrix/Level: <u>Aqueous</u>				
Sample ID: <u>JB48009-1MS/-1MSD</u>	Matrix/Level: <u>Solid</u>				
Sample ID: <u>JB48009-1MS/-1MSD</u>	Matrix/Level: <u>Solid</u>				
Sample ID: <u>JB47867-3MS/-2MSD</u>	Matrix/Level: <u>Solid</u>				
MS OR MSD	COMPOUND	% R	RPD	QC LIMITS	ACTION
<u>JB47460-4MS/-4MSD</u>					
<u>MSD</u>	<u>MIBK</u>	<u>146 %</u>		<u>62-144</u>	<u>No action</u>

Note: No action – criteria apply to the unspiked sample

- * QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.
- * If QC limits are not available, use limits of 70 – 130 %.

Actions:

QUALITY	%R < LL	%R > UL
Positive results	J	J
Nondetects results	R	Accept

MS/MSD criteria apply only to the unspiked sample, its dilutions, and the associated MS/MSD samples:

If the % R for the affected compounds were < LL (or 70 %), qualify positive results (J) and nondetects (UJ).

If the % R for the affected compounds were > UL (or 130 %), only qualify positive results (J).

If 25 % or more of all MS/MSD %R were < LL (or 70 %) or if two or more MS/MSD %Rs were < 10%, qualify all positive results (J) and reject nondetects (R).

A separate worksheet should be used for each MS/MSD pair.

DATA REVIEW WORKSHEETS

All criteria were met X
Criteria were not met
and/or see below _____

VII. B MATRIX SPIKE/MATRIX SPIKE DUPLICATE

MS/MSD – Unspiked Compounds

It should be noted that Region 2 SOP HW-24 does not specify a MS/MSD criteria for the unspiked compounds in the sample. A %RSD of < 50% has therefore been utilized as professional judgment.

If all target analytes were spiked in the MS/MSD, this review element is not applicable.

List the %RSD of the compounds which do not meet the criteria.

Sample ID: _____ Matrix/Level/Unit: _____

COMPOUND	SAMPLE CONC.	MS CONC.	MSD CONC.	% RSD	ACTION
----------	-----------------	----------	-----------	-------	--------

This image shows a single sheet of white paper with horizontal ruling lines. The lines are evenly spaced and run across the width of the page. There are approximately 20 lines visible. The paper appears to be a standard notebook or ledger page.

Actions:

- * If the % RSD > 50, qualify the positive result in the unspiked samples as estimated (J).
* If the % RSD is not calculated (NC) due to nondetected value, use professional judgment to qualify the data.

DATA REVIEW WORKSHEETS

All criteria were met X
Criteria were not met
and/or see below

VIII. LABORATORY CONTROL SAMPLE (LCS) ANALYSIS

This data is generated to determine accuracy of the analytical method for various matrices.

1. LCS Recoveries Criteria

Where LCS spiked with the same analyte at the same concentrations as the MS/MSD?
Yes or No. If no make note in data review memo.

List the %R of compounds which do not meet the criteria

LCS ID	COMPOUND	% R	QC LIMIT
<u>Recoveries within laboratory control limits</u>			

- * QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.
- * If QC limits are not available, use limits of 70 – 130 %.

Actions:

QUALITY	%R < LL	%R > UL
Positive results	J	J
Nondetects results	R	Accept

All analytes in the associated sample results are qualified for the following criteria.

If 25 % of the LCS recoveries were < LL (or 70 %), qualify all positive results (J) and reject nondetects (R).

If two or more LCS were below 10 %, qualify all positive results as (J) and reject nondetects (R).

2. Frequency Criteria:

Where LCS analyzed at the required frequency and for each matrix? Yes or No.

If no, the data may be affected. Use professional judgment to determine the severity of the effect and qualify data accordingly. Discuss any actions below and list the samples affected.

DATA REVIEW WORKSHEETS

All criteria were met X
 Criteria were not met
 and/or see below

IX. FIELD DUPLICATE PRECISION

Sample IDs: JB47485-1/-2

Matrix: Soil

Field duplicate samples may be taken and analyzed as an indication of overall precision. These analyses measure both field and lab precision; therefore, the results may have more variability than laboratory duplicates which only laboratory performance. It is also expected that soil duplicate results will have a greater variance than water matrices due to difficulties associated with collecting identical field duplicate samples.

The project QAPP should be reviewed for project-specific information.

Suggested criteria: RPD \pm 30% for aqueous samples, RPD \pm 50 % for solid samples. If both samples and duplicate are \leq 5 SQL, the RPD criteria is doubled.

COMPOUND	SQL	SAMPLE CONC.	DUPLICATE CONC.	RPD	ACTION
Ethylbenzene	410	41000	10000	122	Qualify results in affected samples (J)
Toluene	160	21200	4810	126	
Xylene (total)	220	159000	40600	119	

Actions:

Qualify as estimated positive results (J) and nondetects (UJ) for the compound that exceeded the above criteria. For organics, only the sample and duplicate will be qualified.

If an RPD cannot be calculated because one or both of the sample results is not detected, the following actions apply:

If one sample result is not detected and the other is greater than 5x the SQL qualify (J/UJ).

If one sample value is not detected and the other is greater than 5x the SQL and the SQLs for the sample and duplicate are significantly different, use professional judgment to determine if qualification is appropriate.

If one sample value is not detected and the other is less than 5x, use professional judgment to determine if qualification is appropriate.

If both sample and duplicate results are not detected, no action is needed.

DATA REVIEW WORKSHEETS

All criteria were met ___N/A___
Criteria were not met
and/or see below _____

IX. LABORATORY DUPLICATE PRECISION

Sample IDs: ____ - _____

Matrix: ____ - _____

Field duplicate samples may be taken and analyzed as an indication of overall precision. These analyses measure both field and lab precision; therefore, the results may have more variability than laboratory duplicates which only laboratory performance. It is also expected that soil duplicate results will have a greater variance than water matrices due to difficulties associated with collecting identical field duplicate samples.

The project QAPP should be reviewed for project-specific information.

Suggested criteria: RPD \pm 30% for aqueous samples, RPD \pm 50 % for solid samples. If both samples and duplicate are <5 SQL, the RPD criteria is doubled.

COMPOUND	SQL	SAMPLE CONC.	DUPLICATE CONC.	RPD	ACTION

Actions:

Qualify as estimated positive results (J) and nondetects (UJ) for the compound that exceeded the above criteria. For organics, only the sample and duplicate will be qualified.

If an RPD cannot be calculated because one or both of the sample results is not detected, the following actions apply:

If one sample result is not detected and the other is greater than 5x the SQL qualify (J/UJ).

If one sample value is not detected and the other is greater than 5x the SQL and the SQLs for the sample and duplicate are significantly different, use professional judgment to determine if qualification is appropriate.

If one sample value is not detected and the other is less than 5x, use professional judgment to determine if qualification is appropriate.

If both sample and duplicate results are not detected, no action is needed.

DATA REVIEW WORKSHEETS

All criteria were met X
Criteria were not met
and/or see below

X. INTERNAL STANDARD PERFORMANCE

The assessment of the internal standard (IS) parameter is used to assist the data reviewer in determining the condition of the analytical instrumentation.

List the internal standard area of samples which do not meet the criteria.

- * Area of +100% or -50% of the IS area in the associated calibration standard.
* Retention time (RT) within 30 seconds of the IS area in the associated calibration standard.

DATE	SAMPLE ID	IS OUT	IS AREA	ACCEPTABLE RANGE	ACTION
------	-----------	--------	---------	------------------	--------

Internal standard area within laboratory control limits

Actions:

1. IS actions should be applied to the compound quantitated with the out-of-control ISs

QUALITY	IS AREA < -25%	IS AREA = -25 % TO - 50%	IS AREA > + 100%
Positive results	J	J	J
Nondetected results	R	UJ	ACCEPT

2. If a IS retention time varies more than 30 seconds, the chromatographic profile for that sample must be examined to determine if any false positive or negative exists. For shifts of a large magnitude, the reviewer may consider partial or total rejection of the data for the sample fraction.

DATA REVIEW WORKSHEETS

All criteria were met X
Criteria were not met
and/or see below

XII. SAMPLE QUANTITATION

The sample quantitation evaluation is to verify laboratory quantitation results. In the space below, please show a minimum of one sample calculation:

JB47485-11MS

Ethylbenzene

RF = 1.626

$$[] = (6454676)(50)/(302052)(1.626)$$

$$= 657 \text{ ppb OK}$$

DATA REVIEW WORKSHEETS

All criteria were met X
Criteria were not met
and/or see below _____

XII. QUANTITATION LIMITS

A. Dilution performed

[illegible]

B. Percent Solids

List samples which have $\leq 50\%$ solids

Actions:

If the % solids of a soil sample is 10-50%, estimate positive results (J) and nondetects (UJ)

If the % solids of a soil sample is < 10%, estimate positive results (J) and reject nondetects (R)